## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Serial No. : 10/511349

Applicant : Eugene et al.

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Title : A Polyurethane Resin Derived from Polyhydroxylated Resins

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Examiner : Gorr

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## **DECLARATION OF GILLES EISELE**

I, Gilles Eiselé, French citizen, do hereby declare:

I am co-inventor of the patent application 10/511349. I am a chemist, engineer from the High School of Chemistry from Mulhouse. I'm graduated (PhD) in Chemistry and Photochemistry from the University of Haute-Alsace (France). I have been working in the filed of printing inks since 11 years as R&D Manager for Polymer Synthesis. Therefore, I consider myself an expert in this field.

I have studied U.S. Patent 6,518,359 to Clemens, which I discuss below. In part I of example 37 to 41 (col. 34, l. 59, to col. 35, l. 9), the preparation of the pre-polymer is described. I added the amounts of the ingredients used in part I, and got a total of 632.74 parts by weight.

Example 37 indicates that only 170 parts by weight of said prepolymer made in part I are used (col. 35, 1. 14). Thus, in order to arrive at the true amounts used in example 37, for the components making up the prepolymer, I divided the total values by 170:632.74/170 = 3.72.

To determine whether free isocyanate groups are left which may be reacted with NIREZ 2019, I calculated the equivalent amounts. According to basic stoichiometry, the equivalent amount is defined by the equation

$$n^{eq} = z * n$$

z being the number of the functional groups of interest in the molecule (i.e. the functional groups which actually react in the reaction of interest), and

n being the molar amount which is defined to be m/M, where m is the amount of a substance used in the reaction, and M being the molar amount of the substance.

Replacing n in the above equation by m/M, one arrives at

$$n^{eq} = z * m/M$$
.

Now, the equivalent mass  $M^{eq}$  of a substance is defined as  $M^{eq} = M/z$ . Thus,  $z/M=1/M^{eq}$ . Substituting z/M in the above equation, one arrives at

$$n^{eq} = m/M^{eq}$$
.

The equivalent weights of the ingredients are known (molecular weight divided by the number of functional groups participating in the reaction):

For IPDI, M<sup>eq</sup>=112 g/mol.

For ACCLAIM 3201 (which is a polypropylene glycol with a molecular weight of 3000), M<sup>eq</sup>=1500 g/mol.

For DMPA, Meq=67 g/mol

For EDA, Meq=31 g/mol

In Example 37, m is the amount in parts by weight used in the examples. Since 170 parts by weight of the prepolymer were used, the partial amounts by weight given in part I of the examples have to be divided by 3.72, as explained above. This calculation yielded the following equivalent amounts  $n^{eq}$ :

0.1895 equivalents isocyanate

0.0623 equivalents OH (from Acclaim 3201)

0.0631 equivalents OH (from DMPA)

0.0617 equivalents NH<sub>2</sub> (from EDA).

Summing the equivalents of the isocyanate-reactive components (Acclaim 3201, DMPA and EDA), I arrived at <u>0.1871 equivalents</u> of isocyanate-reactive components.

This is substantially equal to the number of equivalents of the isocyanate (0.1895) used in the reaction. Taking further into account that a substantial amount of water is added in example 37 (col. 35, 1. 17-20) which of course reacts with any remaining isocyanate groups, in my opinion there would have been virtu-

ally no isocyanate left to react with NIREZ 2019. Indeed, in example 37, at column 35, lines 18 and 19, it is stated that a prepolymer/NIREZ "mixture" is obtained. In a mixture, the components have not reacted with each other, but instead they coexist.

I also noticed that in examples 38 to 41, the amount of isocyanate which was incorporated with the prepolymer from part I was left unamended, while the amount of NIREZ 2019 was significantly increased, in comparison to other examples. To promote a reaction between NIREZ 2019 and isocyanate, one would not have increased the amount of the NIREZ 2019, which was already present in sufficient quantity; rather, the amount of isocyanate would have been increased in order to provide a reaction partner for the NIREZ 2019. Because the opposite is done in examples 38 to 41, I conclude that NIREZ 2019 would not react with the isocyanate, but instead would function as a non-reacting additive.

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that the making of willful false statements or the like is punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patents issued thereon.

Annemasse, 2<sup>nd</sup> october 2006

Gilles Eisele